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# Thermal Stability of the Microstructure of an Aged Nb-Zr-C Alloy

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Work performed for

**U.S. DEPARTMENT OF ENERGY**  
**Nuclear Energy**  
**Reactor Systems Development and Technology**

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## SUMMARY

The effects of thermally aging with and without an applied stress on the microstructure of a Nb-Zr-C alloy containing 0.9 wt % Zr and 0.06 wt % C were studied. Chemical analysis, metallographic examination, energy dispersive x-ray spectra of the bulk material, and chemical and x-ray analyses of the phase-extracted residue were used to characterize the microstructure. The samples examined were from a creep strength study involving hot and cold working, and various combinations of exposure to temperatures ranging from 1350 to 1755 K with and without applied load for times as long as 34 000 plus hours. The results showed that the initial microstructure consisted primarily of orthorhombic precipitates of Nb<sub>2</sub>C which were partially or completely transformed to face-centered cubic carbides of Nb and Zr, (Zr, Nb)C, upon prolonged exposure to elevated temperatures. Furthermore, it was found that the microstructure of the alloy is extremely stable owing to the very finely distributed precipitates throughout its matrix and along the grain boundaries. The lattice parameters of the cubic carbides were determined to vary from 0.458 to 0.465 nm as the Zr/Nb ratio varied from 38/62 to 75/25.

## INTRODUCTION

A material is selected for an application based on its properties which are often strong functions of chemical composition and microstructure. Inasmuch as the microstructure, hence the properties may be altered markedly by processing, the characterization of the microstructure as it relates to the properties and processing of an engineering material is of significant importance.

Niobium-based alloys, like other refractory metal alloys, are potential candidates for high temperature use. Nb-1 wt % Zr and Nb-1 wt % Zr-C alloys have been identified as the materials for fuel cladding and structural components in space nuclear power applications (Cooper 1984; Kruger et al. 1987; Dokko et al. 1984). These applications will require high toughness and good creep resistance at temperatures of about 1250 to 1450 K for times of 7 years or more. Nb-1%Zr-C alloys are shown to have a creep strength far

better than Nb-1%Zr (Titran 1986; Titran et al. 1987; Titran 1990), good weldability and weld strength (Moore et al. 1986; Titran et al. 1986), a relatively stable microstructure (Grobstein and Titran 1986), and much higher strength than either Nb-1%Zr or Nb-C (Delgrosso et al. 1967; Grigorovich and Sheftel 1982).

A number of studies have been published on the analysis and characterization of the microstructure of Nb-Zr-C alloys (Arzamosov and Vasil'eva 1978; Begley et al. 1963; Cuff 1962; Delgrosso et al. 1967; Grobstein and Titran 1986; Jun and Hoch 1971; Khandarov et al. 1978; Kissil' et al. 1976; Korotayev et al. 1980, 1981; Lyutyi et al. 1978; Maximovich et al. 1978; Moiseev et al. 1983; Norton and Mowry 1949; Ostermann 1971; Ostermann and Bollenrath 1966; Tsvikilevich 1980). In all these studies, there is general agreement that the high toughness and the excellent creep resistance of the Nb-Zr-C alloys are attributable to the presence of stable carbides of Nb and/or Zr along the grain boundaries as well as in the matrix. These are reported to be nonstoichiometric carbides of Nb and Zr,  $(\text{Nb}, \text{Zr})\text{C}_x$ , or solid solutions of NbC and ZrC with lattice parameters varying directly with the Zr/Nb ratio (Jun and Hoch 1971; Moiseev et al. 1983; Norton and Mowry 1949; Ostermann 1971).

The work reported herein is part of an ongoing project of a critical evaluation of Nb-Zr-C alloys at NASA Lewis Research Center, Cleveland, Ohio, and deals with the characterization of the microstructures of Nb-0.9 wt % Zr-0.06 wt % C alloy samples with different processing histories including creep testing for as long as about 34 500 hr under a 10 MPa stress at 1350 K. It should be noted that this is the first paper dealing with samples subjected to elevated temperatures for more than 10 000 hr.

## EXPERIMENTAL

### Materials

The samples examined in this study were from a Nb-0.9%Zr-0.06%C alloy obtained from Oak Ridge National Laboratory in the form of as-rolled 1-mm thick sheet. All the samples were given a double-anneal (DA) treatment, (1 hr at 1755 K + 2 hr at 1475 K). They were then subjected to some combination of heat treatment and creep testing with or without prior aging for 1000 hr at 1350 or 1400 K. The samples studied were the following: as-rolled sheet, DA, DA + 1000 hr at 1350 K, DA + 34 500 hr at 1350 K, DA + 34 500 hr at 1350 K under 10 MPa stress, DA + 1329 hr at 1400 K, DA + 1531 hr at 1350 K, DA + 33 500 hr at 1350 K, DA + 1000 hr at 1350 K + 32 500 hr at 1350 K under 10 MPa stress. All the creep testing and heat treatments were conducted in a chamber under a pressure of the order of about  $10^{-7}$  Pa. The samples were wrapped in a chemically-cleaned tantalum foil prior to creep testing or heat treatment.

### Procedure

To monitor the loss or pick up of the interstitial solutes, each sample was analyzed for oxygen, nitrogen and carbon following creep testing or heat treatment.

All the specimens were examined in the as-polished condition with the light microscope. In an attempt to more clearly define the microstructure, some samples were examined in the interference layered, anodized, or stain-etched conditions. Some samples were etched with a solution of 30 lactic acid - 15 nitric acid - 5 hydrofluoric acid (by volume) in a stepwise fashion (a few seconds at a time) so as not to lose the precipitates as warned against in an earlier publication (Grobstein and Titran, 1986), and the results were fairly successful. Several samples also were examined with the scanning electron microscope in the as-polished condition, with limited success, however.

A solution of 900 ml methanol - 100 ml bromine - 10 g tartaric acid was used for phase extraction with platinum wire wrapped around the sample as the catalyst. The solution was vibrated ultrasonically twice a day, and the process for most samples was continued for varying times until all the bulk alloy was dissolved. The solution then was filtered through a 0.22- $\mu$ m Teflon filter paper, and the residue was washed with alcohol and dried in an oven at about 130 °C. The Nb and Zr contents of the phase extracted residues were determined by inductively-coupled plasma technique (ICP). X-ray analysis was used to determine the crystal structures and lattice parameters of the residues. To check the results obtained from the analysis of the phase-extracted residue, an energy dispersive x-ray spectra (EDS) analysis was performed on precipitates of different sizes in some samples using scanning electron microscope (SEM).

## RESULTS AND DISCUSSION

### Chemical Analysis

The chemical analysis results of the samples examined in this study can be seen from table 1. The end portions of the creep-tested specimens are considered only heat-treated since they do not experience any significant load during testing. These specimens are 13E, 15E, 28E, and 34E. As will be noted from the table, the material may have lost some oxygen and carbon upon double annealing as well as aging. The loss may be as CO and/or CO<sub>2</sub> formed during this heat treatment. This is not the case, however, for creep-tested samples which show an overall gain of oxygen possibly due to contamination from the experimental environment. Interesting to note is that the oxygen content of the middle portions of these samples (13M, 15M, 28M, 34M) are considerably higher than that of the end portions. This may be due to the oxygen contamination from the chamber, and its subsequent thermotransport from the cold to the hotter regions of the sample as the ends of the creep samples were reported to be 20 to 30 °C colder than the middle. Such thermotransport was reported for carbon in Nb-Zr-C solid solution alloys, and the thermotransport behavior of oxygen is often similar (Uz and Schmidt 1989). The lack of oxides in metallographic examination and the phase-extracted residues of the samples would indicate that all the oxygen was in solid solution at the temperatures of interest. The suggestion that the higher oxygen content in the middle may arise from the increased mobility of oxygen under stress (Grobstein and Titran 1986) is another possibility, however, the total strain in the samples tested for 30 000 plus hours was determined to be zero.

TABLE 1. Chemical Compositions of Samples from Nb-0.9%Zr-0.06%C Alloy

Sample ID	Heat Treatment			Chemical Analysis			
	Stress (MPa)	t (hr)	T (K)	O <sup>a</sup> , (ppmw)	N <sup>a</sup> (ppmw)	C <sup>b</sup> (ppmw)	Zr <sup>c</sup> (wt %)
As-rolled		-----	-----	-----	78	53	630
0.9							
DA	--	1	1755				
		2	1475	34	50	500	
Age	--	1 000	1350	31	30	600	--
13E <sup>d</sup>	--	1 329	1400	14	41	600	--
13M <sup>d</sup>	--	1 000	1400				
	35	329	1400	320	41	600	--
15E	--	1 531	1350	200	50	600	--
15M	--	1 000	1350				
	40	531	1350	313	60	--	--
28E	--	34 500	1350	80	32	640	--
28M	10	34 500	1350	430	26	620	--
34E	--	33 500	1350	120	40	640	--
34M	--	1 000	1350				
	10	32 500	1350	270	35	610	--

<sup>a</sup>Inert gas fusion method.

<sup>b</sup>Combustion extraction method.

<sup>c</sup>Inductively-coupled plasma method.

<sup>d</sup>(E) refers to the stress free area where (M) refers to the stressed area of crept specimens.



## Metallography

The microstructure of the as-rolled material consisting of relatively coarse precipitates ( $>1\text{ }\mu\text{m}$ ) aligned in the rolling direction, can be seen from figure 1. Figure 2 shows the microstructure of a sample that was double-annealed (DA) and of one that was also aged (DA + 1000 hr at 1350 K). These show that full recrystallization took place upon double-annealing, and that the microstructure did not coarsen upon subsequent aging. Relatively fine distribution of the precipitates can be seen from the stain-etched micrograph of the aged specimen in figure 3. The microstructures of the samples heated for  $>30\text{ }000\text{ hr}$  (#28 and #34 in table 1) were examined in the as-polished condition optically and also by SEM. Optical examination was also made on some samples in the etched and anodized conditions. The microstructures of the end and the middle portions of these samples were similar. Typical microstructures of these specimens, figure 4, show very little, if any coarsening as compared to the double-annealed and/or aged microstructures. The excellent stability of the Nb-0.9%Zr-0.06%C alloy can be attributed to the fine distribution of precipitates along the grain boundaries and throughout the matrix. An extremely fine distribution of the submicron-sized precipitates can also be seen in the SEM micrographs of the 34 500 hr creep sample, figure 5 (#28 in table 1). The above observations would indicate that the thermomechanical treatment gives rise to a refinement of the microstructure, and that prolonged heat treatment results in the dissolution of some coarser precipitates, and/or formation of very finely-distributed precipitates throughout the matrix.

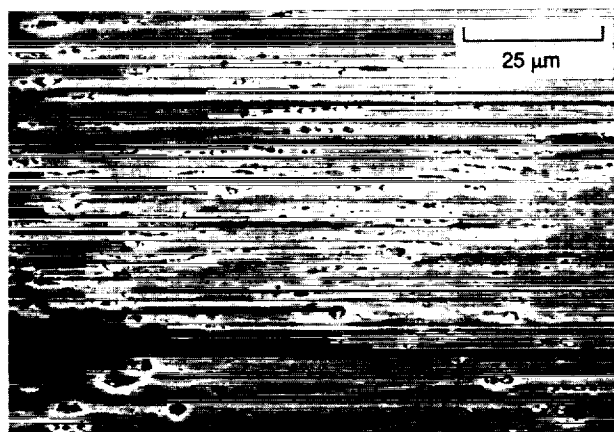
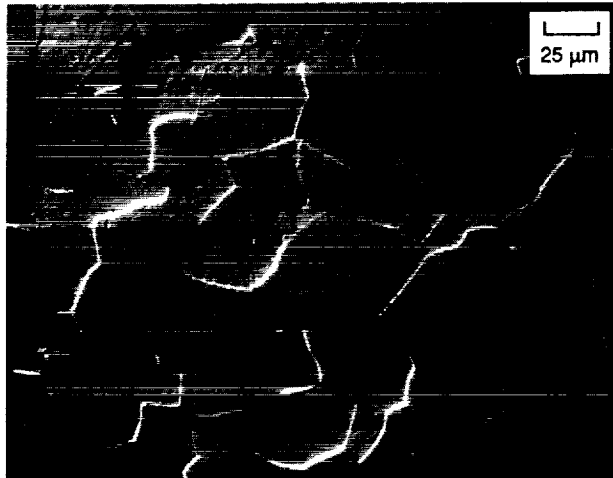


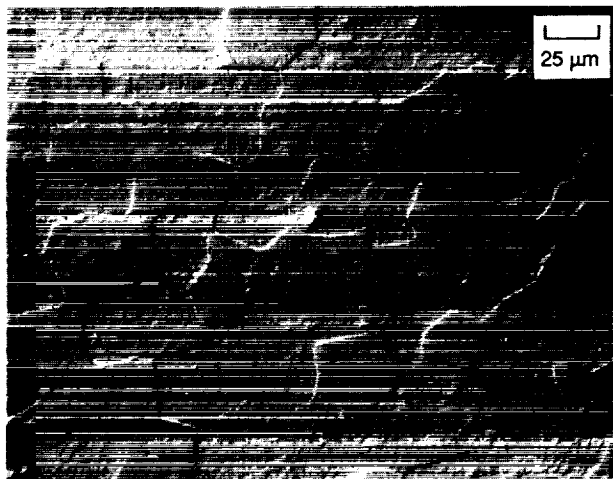
Figure 1.—SEM micrograph of Nb-0.9% Zr-0.06% C alloy in the as-rolled condition. As-etched, 10 000X.

## Microstructure Characterization

The characterization of the microconstituents of the Nb-0.9%Zr-0.06%C alloy was attempted by analyzing the phase-extracted residue from a number of samples and by employing energy dispersive x-ray analysis in SEM.



(a) Double-annealed.



(b) Double-annealed and aged for 1000 h at 1350 K. As-etched, 400X, DIC.

Figure 2.—Optical micrographs of samples from Nb-0.9% Zr-0.06% C alloy.

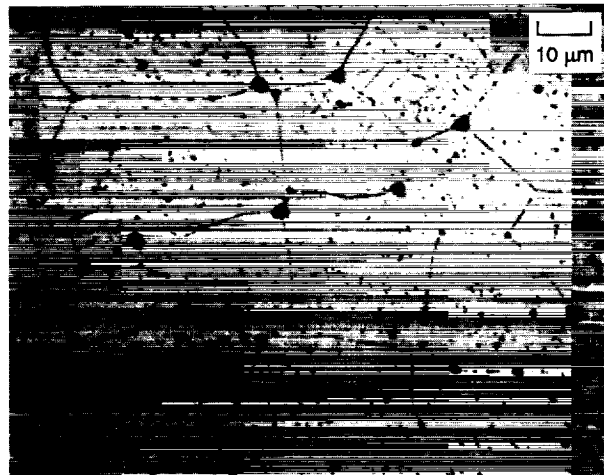
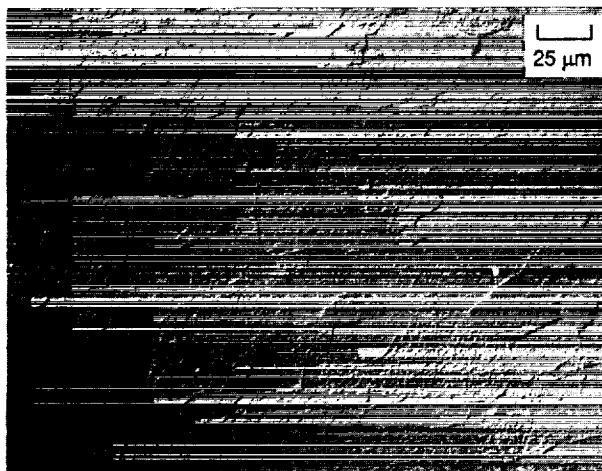


Figure 3.—Optical micrograph of a sample from Nb-0.9% Zr-0.06% C alloy as double-annealed and aged for 1000 h at 1350 K. As stain-etched, 1000X, DIC.

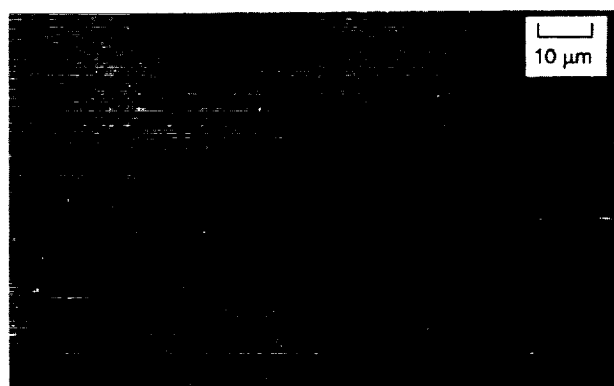


(a) Double-annealed and heat-treated for 34 500 h, (28E), as-etched.

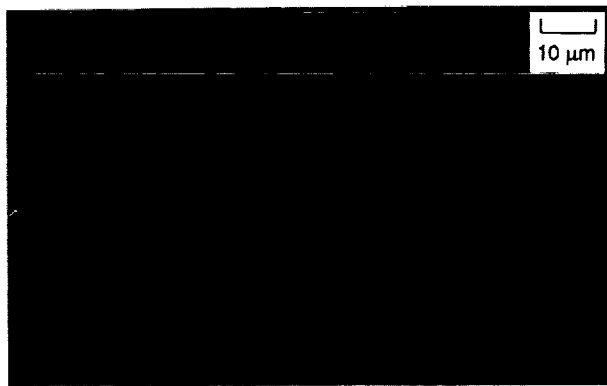


(b) Double-annealed and aged for 1000 h at 1350 K and then creep tested at 1350 K for 32 500 h under 10 MPa stress, (34M), as-polished and anodized.

Figure 4.—Optical micrographs of samples from Nb-0.9% Zr-0.06% C alloy. Both 400X, DIC.



(a) 1000X.



(b) 10 000X.

Figure 5.—SEM micrographs of a sample from stress free area of Nb-0.9% Zr-0.06% C alloy (28E). Double-annealed and creep-tested at 1350 K for 34 500 h under 10 MPa stress. Both as-polished, secondary-electron image.

### Phase Extraction

The phase-extracted residue from each sample was analyzed for Nb and Zr content, crystal structure, and lattice parameters. The results obtained are tabulated in table 2. As will be noted from the results of #13E and #15E, the length of the phase extraction process had a marked effect on the Nb and Zr contents of the residue. This may be due to insufficient time for all Nb from the matrix to dissolve, or to some of the Zr in the carbides being attacked upon prolonged processing, or possibly to the chemical analysis techniques and procedures used. Even though phase extraction has been in use for over two decades, no study of the possible effects of processing variables such as time, metal-to-solution ratio, amount of catalyst, and procedures has been reported.

The results obtained show that the precipitates are predominantly  $\text{Nb}_2\text{C}$  in the rolled and double-annealed Nb-0.9%Zr-0.06%C alloys, and that prolonged heat treatment results in the conversion of the orthorhombic  $\text{Nb}_2\text{C}$  to a face-centered cubic carbide of Nb and Zr,  $(\text{Nb}, \text{Zr})\text{C}$ , or to NbC and ZrC and the subsequent formation of a solid solution between them. The lattice parameters determined in this study fall between the values for NbC and ZrC as marked in table 3 which gives the crystal structures and the lattice parameters of the precipitates that may exist in the alloy. The general trend is that the Zr/Nb ratio increases with the length of the heat treatment accompanied by an increase in the lattice parameter. This is as expected since Zr has a larger atomic radius (0.158 nm) than Nb (0.143 nm), and the lattice parameter of ZrC (0.470 nm) is considerably greater than that of NbC (0.447 nm).

TABLE 2. Results of Phase Extraction Studies of Samples From Nb-0.9%Zr-0.06%C Alloy

Sample ID	Time (h)	Phases Present	Lattice Parameter <sup>a</sup>	
			$a_o$ (nm)	Zr/Nb <sup>b</sup> Ratio
As-rolled	--	Nb <sub>2</sub> C+cubic	4.68	0.7/99.3
DA	--	Nb <sub>2</sub> C+cubic	4.58	4.1/95.9
AGE	--	Cubic	4.60	38/62
13E	--	Cubic	4.61	77/23
		Cubic	4.64	77/23
13E	17	Cubic	4.61	56/43
13M	--	Cubic	4.62	70/30
15E	--	Cubic	4.62	73/27
15E	--	Cubic	4.60	46/53
15E	88	Cubic	4.62	57/42
15M	-	Cubic	4.61	68/32
28E	330	Cubic	4.65	72/27
28M	330	Cubic	4.64	63/37
34E	330	Cubic	4.65	60/39
34M	330	Cubic	4.65	55/44

<sup>a</sup>X-ray spectrometry.<sup>b</sup>Inductively-coupled plasma technique.

In an earlier study on different samples from this same alloy, Grobstein and Titran (1986) discussed the possibility of the formation of ZrO<sub>2</sub> and ZrO. Existence of ZrO<sub>2</sub> in this material would not be likely as the lattice parameters obtained are much smaller than that of ZrO<sub>2</sub>. Proving the existence or absence of ZrO is not possible with the methods used since its lattice parameter falls within the range of values obtained in this work. ZrO could be present at the middle portions of the samples #28 and #32 due to the considerably higher oxygen contents therein (table 1), and its presence would be signaled by a difference in the lattice parameter. The lattice parameters of the ends and the middle portions of these specimens were the same (table 2), however, in spite of the large difference in their oxygen contents.

The results obtained in this study suggest that the precipitates which impart the Nb-0.9%Zr-0.06%C alloy its excellent microstructural stability, and hence, its excellent creep resistance are carbides of Nb and Zr. These carbides have a face-centered cubic structure with  $a_o$  values between 0.458

TABLE 3. Crystal Structures and Lattice Parameters of Compounds Possibly Present in Nb-Zr-C Alloys

Compound	Crystal structure	Lattice Parameter (nm)		
		$a_o$	$b_o$	$c_o$
Nb <sub>2</sub> C	Hexagonal	0.313	----	0.497
Nb <sub>2</sub> C	Orthorombic	1.09	0.31	0.50
NbO	Cubic	0.421	----	-----
NbN	Tetragonal	0.438	----	0.431
NbN	Hexagonal	0.295	----	0.553
NbC	fcc(B1)	→ 0.447	----	-----
ZrN	fcc(B1)	0.456	----	-----
ZrO	fcc(B1)	0.462	----	-----
ZrC	fcc(B1)	→ 0.470	----	-----
ZrO <sub>2</sub>	fcc(C1)	0.509	----	-----

to 0.468 nm with an accompanying change in the Zr/Nb ratio from about 35/65 to 75/25. These results are consistent with those reported in the literature. June and Hoch (1971), Norton and Mowry (1949) and Moiseev and Vatolin (1983) all report similar results for the  $a_o$  values and the corresponding Zr/Nb ratios. They also indicate that the solid solutions of NbC and ZrC yield nonstoichiometric carbides of (Zr, Nb)C<sub>x</sub> with x varying from 0.8 to 0.98, or Zr<sub>x</sub>Nb<sub>1-x</sub>C<sub>y</sub> with y varying again between 0.8 and 0.98.

#### Energy-Dispersive X-Ray Spectrometry:

Energy-dispersive X-ray spectrometry (EDS) was performed on some of the as-polished samples using SEM. The results of the EDS analyses can be seen from table 4. The results from the larger precipitates (>1  $\mu$ m) are in good agreement with those obtained from the analysis of the phase-extracted residue (table 2), but a decrease in the Zr/Nb ratio is observed with decreasing precipitate size. This is attributable to the small size of the precipitates giving rise to the sampling of the matrix during EDS analysis. Noted also from table 4 is the Zr/Nb ratio obtained from the matrix. Even though the alloy contains 0.9%Zr, the EDS analysis of the matrix repeatedly yielded 4 to 5%Zr. This error is thought to be due to sampling errors and our inability to correct the spectra due to the lack of standards of the proper size and composition.

TABLE 4. EDS Analysis Results of  
Precipitates in Samples From  
Nb-0.9%Zr-0.06%C Alloy

Sample ID	Precipitate size ( $\mu\text{m}$ )	Zr/Nb <sup>a</sup> ratio
AGE	--	53/57
28M	>1	65/35
28M	0.5	47/53
28M	0.1-0.2	10/90
28M	matrix	5/95
34M	1-2	75/25
34M	1	66/34
34M	0.5	48/52
34M	0.3	27/73
34M	0.1-0.2	11/89
	matrix	4/96

<sup>a</sup>Spectral analysis uncorrected.

#### SUMMARY OF RESULTS

The effects of thermomechanical treatment on the microstructure of a Nb-0.9%Zr-0.06%C alloy was investigated. The results obtained are as follows:

1. In the rolled, double-annealed and/or aged conditions, the microstructure predominantly consists of relatively coarse ( $>1 \mu\text{m}$ ) orthorhombic carbides of  $\text{Nb}_2\text{C}$ .
2. Prolonged exposure of the material to elevated temperatures with or without applied stress gives rise to the conversion of  $\text{Nb}_2\text{C}$  particles to fine face-centered cubic carbides of Nb and Zr, very finely distributed throughout the matrix.
3. The carbides of Nb and Zr, (Nb, Zr)C have a lattice parameter ranging from about 0.458 to 0.468 nm with an accompanying change in the Zr/Nb ratio from about 35/65 to 75/25.
4. The material has excellent microstructural stability at temperatures as high as 1350 K with or without an applied stress of 10 MPa for times well over 30 000 hr owing to the presence of stable (Nb, Zr)C particles very finely distributed throughout its matrix.

## CONCLUSIONS

The excellent long time, high temperature creep strength of the Nb-0.9%Zr-0.06%C alloy is due to the thermal stability of its (Zr,Nb)C carbide precipitates and microstructure. A critical study of the characterization and analysis techniques is advisable. A systematic study of the Nb-Zr-C alloys is necessary to establish the processing-microstructure-properties relationship for these materials.

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16. Abstract The effects of thermally aging with and without an applied stress on the microstructure of a Nb-Zr-C alloy containing 0.9 wt % Zr and 0.06 wt % C were studied. Chemical analysis, metallographic examination, energy dispersive x-ray spectra of the bulk material, and chemical and x-ray analyses of the phase-extracted residue were used to characterize the microstructure. The samples examined were from a creep strength study involving hot and cold working, and various combinations of exposure to temperatures ranging from 1350 to 1755 K with and without applied load for times as long as 34 000 plus hours. The results showed that the initial microstructure consisted primarily of orthorhombic precipitates of Nb <sub>2</sub> C which were partially or completely transformed to face-centered cubic carbides of Nb and Zr, (Zr, Nb)C, upon prolonged exposure to elevated temperatures. Furthermore, it was found that the microstructure of the alloy is extremely stable owing to the very finely distributed precipitates throughout its matrix and along the grain boundaries. The lattice parameters of the cubic carbides were determined to vary from 0.458 to 0.465 nm as the Zr/Nb ratio varied from 38/62 to 75/25.					
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